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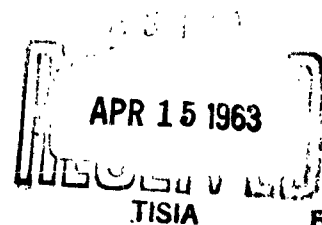
Report No. 8926-129

Material - Adhesives - Structural - EC-1459, EC-1469, AF-102
(Minnesota Mining and Manufacturing Co.)

Effect of Variations in Bonding Pressures and
Temperatures on Joint Strengths

R. W. Lawley, Jr., M. C. Miyaji, W. M. Sutherland

9 July 1957



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Material - Adhesives - Structural - EC-1459, EC-1469, AF-102
(Minnesota Mining and Manufacturing Co.)

Effect of Variations in Bonding Pressures and
Temperatures on Joint Strengths

Abstract:

The effects of variations in pressures and temperatures for curing adhesive bonded joints made between 2024-T86 aluminum alloy overlaps, and 2024-T86 aluminum alloy face plates and 3003-H19 aluminum alloy honeycomb cores with EC-1459, EC-1469 and AF-102 (Minnesota Mining and Manufacturing Co.) adhesives were determined. The curing cycles used were (1) 150 psi pressure at 350°F for 2 hours, (2) 100 psi pressure at 250°F for 30 minutes followed by 350°F for 2 hours, (3) 100 psi pressure at 350°F for 2 hours, and (4) 85 psi pressure at 350°F for 2 hours. The first two of the four cure cycles gave satisfactory -67°F tensile shear strengths. The 216°F tensile shear strength obtained by the last three cure cycles were slightly low. The metal to metal peel strength produced by all four cycles was satisfactory. The honeycomb sandwich flexural strengths at -67°F were satisfactory throughout. The room temperature peel strengths of the honeycomb sandwich varied considerably and only the second cure cycle produced satisfactory strengths.

Reference: Lawley, R. W. Jr., Miyaji, M. C., Sutherland, W. M.,
"Effect of Various Cure Cycles on Metal to Metal and
Metal to Core Bonds Using Adhesive System Detailed in
Specification 8-01318," General Dynamics/Convair,
Report MP 57-461, San Diego, California, 9 July 1957
(Reference attached).

SAN DIEGO

STRUCTURES & MATERIALS LABORATORIES

REPORT 57-461

DATE 7-9-57

MODEL F-106

TITLE

REPORT NO. 57-461

**TITLE: EFFECT OF VARIOUS CURE CYCLES ON
METAL TO METAL AND METAL TO CORE
BONDS USING ADHESIVE SYSTEM DETAILED
IN SPECIFICATION 8-01318**

MODEL: F-106

CONTRACT NO. AF33(600)-30169

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NO. OF DIAGRAMS 8

REVISIONS

[illegible]

ANALYSIS
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REVISED BY

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REPORT NO. 57-461
EFFECT OF VARIOUS CURE CYCLES ON
METAL TO METAL AND METAL TO CORE BONDS
USING ADHESIVE SYSTEM DETAILED IN
SPECIFICATION 8-01318

INTRODUCTION:

Some F-106 parts are being subcontracted. Cure cycles being employed by the Subcontractor differ from the requirements of F-106 bonded parts as outlined in Specification 8-01318. It is the desire of the Subcontractor to employ their established cure cycles in the production of the Delta Dart bonded parts. Because of this difference, a test program was initiated to determine whether alternate cure cycles might be employed. The strength values of bonded test specimens will be used as the criteria in determining the adequacy of the alternate cure cycles.

OBJECT:

To obtain resultant strength data of adhesives (detailed in Specification 8-01318) processed by four different alternate cure cycles.

CONCLUSIONS:

1. The first two of the four cure cycles investigated give satisfactory -67°F tensile shear values. These were in excess of the 2025 psi requirements of Convair Specification O-03007 referenced in Specification 8-01318.
2. The $+216^{\circ}\text{F}$ tensile shear values of all the cure cycles investigated, except the first one, (results shown in Table I) appear to be slightly low. Even though there are no specific requirements called out in Convair Specification O-03007 for 216°F testing, an estimate of probable strength at this temperature can be made by interpolating the room temperature and $+300^{\circ}\text{F}$ requirements. The required strength at 216°F is estimated to be 2450 psi.
3. The room temperature metal to metal peel strength values also exceed the 10 lbs./in. width requirement of Specification O-03007, when the adhesive is cured with alternate cure cycles.
4. The sandwich honeycomb flexural strength values obtained at -67°F and $+216^{\circ}$ were in excess of the 50,000 psi minimum skin stress requirement of Specification 8-01318 for both of the cure cycles investigated.
5. Room temperature peel strengths of the sandwich honeycomb specimens varied considerably and only the second cure cycle investigated (see Table VI) gave values acceptable to the 175 inch lb./3" width requirement of Convair Specification 8-01318.

ANALYSIS

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DESCRIPTION OF SPECIMENS:**1. Metal to Metal Bonds:**

The tensile shear specimens were 1" wide by 1/2" overlap made in accordance with Air Force Specification MIL-A-8431. The material used for the tensile shear specimens was .063" x 4" x 7" 2024-T3 clad aluminum alloy. Peel specimens consisted of two .020" x 1" x 9" clad 2024-T86 aluminum alloy strips. Cleaning and bonding, except for the cure cycles employed, followed the process outlined in Specification O-03007.

2. Metal to Core Bonds:

Sandwich honeycomb specimens, 3" x 16" were cut from 18" x 18" panels bonded in accordance with Specification O-03006 except for the following:

- a. Perforated core (vented to the atmosphere) was used.
- b. Core cleaned by "box spraying" with aliphatic naptha only.
- c. Cure cycles modified as noted in Procedure.

3. The materials used in making the sandwich honeycomb panels were as follows:

- a. Skins: .020" x 18" x 18", 2024-T86 clad aluminum alloy.
- b. Core: .375 thick x 18" x 18", 3003 H19 aluminum alloy, perforated foil .002" thick, 1/8" hex. cell with a density of 8.1 lbs./cu.ft.
- c. Adhesives (all products of Minnesota Mining and Manufacturing Co.):
EC 1459 Primer, EC 1469 Liquid Adhesive, AF 102 Supported Film Adhesive.

TEST PROCEDURE:

The various cure cycles investigated were as follows:

1. Metal to Metal Bonds

- a. Pressure: 150 psi

Heat Cycle: Heat from room temperature to 350° F in 25 minutes and cure at 350° F for 2 hours.

This was accomplished by inserting the samples to be bonded in a thermostatically controlled K & M Laboratory press at room temperature, applying the required pressure, and permitting the press to heat "naturally" to 350° F. They were then cured for the required time at temperature.

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TEST PROCEDURE: (Cont'd)

1. Metal to Metal Bonds (Cont'd)

b. Pressure: 100 psi

Heat Cycle: Heat from room temperature to 250° F in 2.6 minutes maximum and hold for 30 minutes at temperature. Heat to 350° F at a rate of 10° F./min. maximum and cure for 2 hours at temperature. The method employed for this cure cycle consisted of the following:

- (1) The K & M press was first heated to 200° F.
- (2) The specimens were inserted in the hot press, the press was immediately turned up to 250° F., and the required pressure was applied. To insure accuracy in reaching 250° F in a maximum of 2.6 minutes; a dummy sample was first run with a thermocouple imbedded in the glue line. The actual time required to reach 250° F from room temperature was thus established as 2.5 minutes. The specimens were then run at 250° F for the 30 minutes required.
- (3) The press was then turned up to 350° F and the natural heating rate of the press raised the specimen temperature to 350° F at a rate of 10° F./minute.
- (4) The samples were then cured for 2 hours at 350° F.

c. Pressure: 100 psi

Heat Cycle: Room temperature to 350° F at a rate of 4° F./minute maximum, and cure at 350° F for 2 hours.

By connecting a thermocouple lead from the center platen of the K & M press to a Leeds & Northrup Speedomax temperature recorder controller it was possible to control the rate of heating from room temperature to 350° F at less than 4° F./minute (3.4° F./minute actual). As in the previous cure cycles, a dry run was made first to prove out the heat rate employed.

d. Pressure: 85 psi

Heat Cycle: Room temperature to 350° F at a maximum rate of 4° F./minute maximum and cure at 350° F for 2 hours. The method employed for this cure cycle was identical to that used in the previous one.

2. Sandwich Honeycomb Bonding Cycles:

Only two cure cycles were investigated for sandwich honeycomb. Three 18" x 18" panels were made up so that one could be used as a temperature survey unit. This panel had a thermocouple imbedded in its center so that actual temperature rise in the test parts could be established. Sandwich honeycomb bonding was accomplished in the black brothers, thermostatically controlled,

TEST PROCEDURE: (Cont'd)

2. Sandwich Honeycomb Bonding Cycles: (Cont'd)

hydraulic press, located in the Adhesive Laboratory. The two cure cycles investigated were:

a. Pressure: 100 psi

Heat Cycle: Heat from room temperature to 250° F in 2.6 minutes maximum and hold for 30 minutes. Increase temperature to 350° F at a maximum rate of 10° F./min. and cure for 2 hours at temperature.

Since this cure cycle is identical to one of those used with the metal to metal test samples, it was hoped that the identical procedure could be used. Unfortunately, because of the greater mass of the 18" x 18" sandwich honeycomb specimen, it was not possible to do this. However, only a slight modification was necessary. Instead of inserting the specimen into the press after it was heated to 200° F it was necessary to have the platens at 250° F. The sandwich panel then came up to 250° F in 2 minutes and 10 seconds. It was then cured at 250° F for 30 minutes. Because of the slower natural heating rate of the Black Brothers press, the temperature rise from 250° to 350° F was 6 1/2° F./minute, which was slightly slower than the 10° F./minute rise used for the metal to metal specimens. The sandwich was then cured for the 2 hours required at 350° F.

b. Pressure: 100 psi

Heat Cycle: Room temperature to 350° F at a maximum rate of 4° F./min. and cure at temperature for 2 hours. The same system was used with this panel as was used with the corresponding metal to metal cure cycle. The Speedomax temperature recorder-controller was connected to a thermocouple imbedded in the sandwich and the temperature rise was controlled by it.

3. Testing:

a. Metal to Metal Specimens:

After bonding, the metal to metal specimens were cut into 1" wide tensile shear specimens and the edges were filed smooth. Testing was accomplished in a circulating air chamber cooled to -67° F for the low temperature evaluations, and the chamber heated to +216° F for the elevated temperature determinations. This chamber was mounted in a Baldwin Southwark Universal Testing Machine and tensile shear strengths were determined with this machine. The peel specimens were tested at room temperature on a rotating drum type machine at approximately 10"/minute in accordance with Convair Report No. ZM393D.

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TEST PROCEDURE: (Cont'd)

3. Testing: (Cont'd)

b. Sandwich Honeycomb Specimens:

The 18" x 18" sandwich honeycomb panels were cut into 3" x 16" specimens with the core ribbon direction running lengthwise to the 16" direction. Flexural tests were conducted on these specimens using the 2 point loading system according to the method outlined in Convair Report No. ZM393D at -67° F and 216° F. The undamaged portions of the flexural samples were cut into 2" wide peel specimens and tested on the rotating drum type peel machine at room temperature.

RESULTS:

The results of these tests are shown in Tables I through VI and Figures I and II.

DISCUSSION OF RESULTS:

The first cure cycle employed for the metal to metal bonds is essentially the same as is in Specification O-03007, except for the pressure used. The results were satisfactory and are relatively easy to obtain.

The second cycle investigated however, posed some problems, both with the metal to metal and the sandwich honeycomb specimens. First it was necessary to place the specimen in the hot presses to obtain a fast enough heating rate from room temperature to 250° F. The pressure had to be applied simultaneously and this proved to be a difficult operation. "Gassing" of the adhesive and subsequent "bubbling" probably occurred because of the fast heating rate, and the lower metal to metal tensile shear results are probably a result of this. It cannot be explained why the sandwich honeycomb flexural values are so high for this cure cycle, but the low peel results are a result of "bubbling" of the adhesive caused by the rapid heating rate.

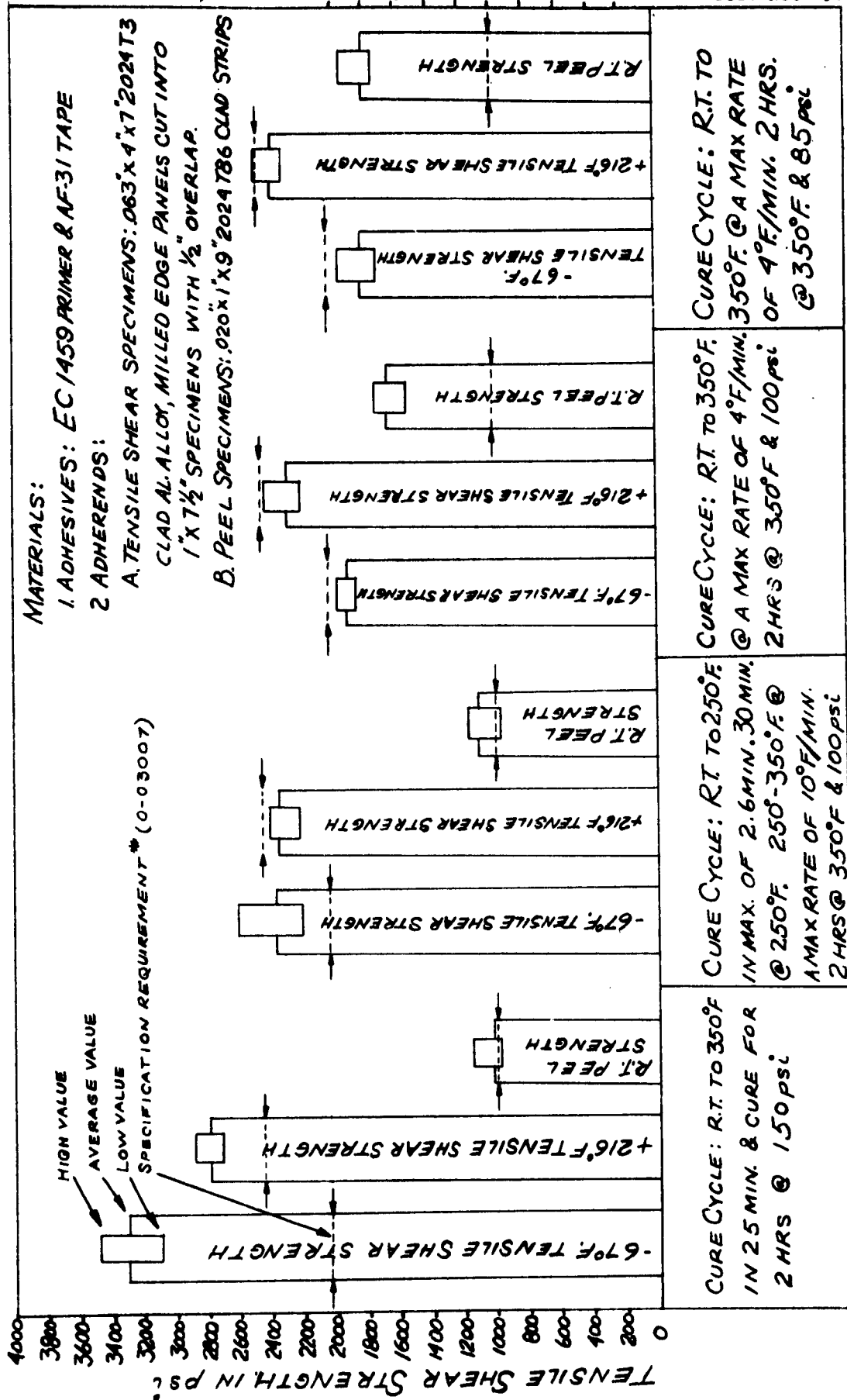
The third cure cycle investigated gave a good combination of sandwich honeycomb flexural and peel results, and perhaps the low tensile shear values could be improved by further testing. The metal to metal peel results were also acceptable as is.

The metal to metal values obtained in the fourth cure cycle were essentially the same as those resulting from the third cure cycle. It does not appear that the 15 psi decrease in curing pressure had much effect on the results.

NOTE:

The test data presented in this report are recorded in the Engineering Test Lab Notebook No. 989.

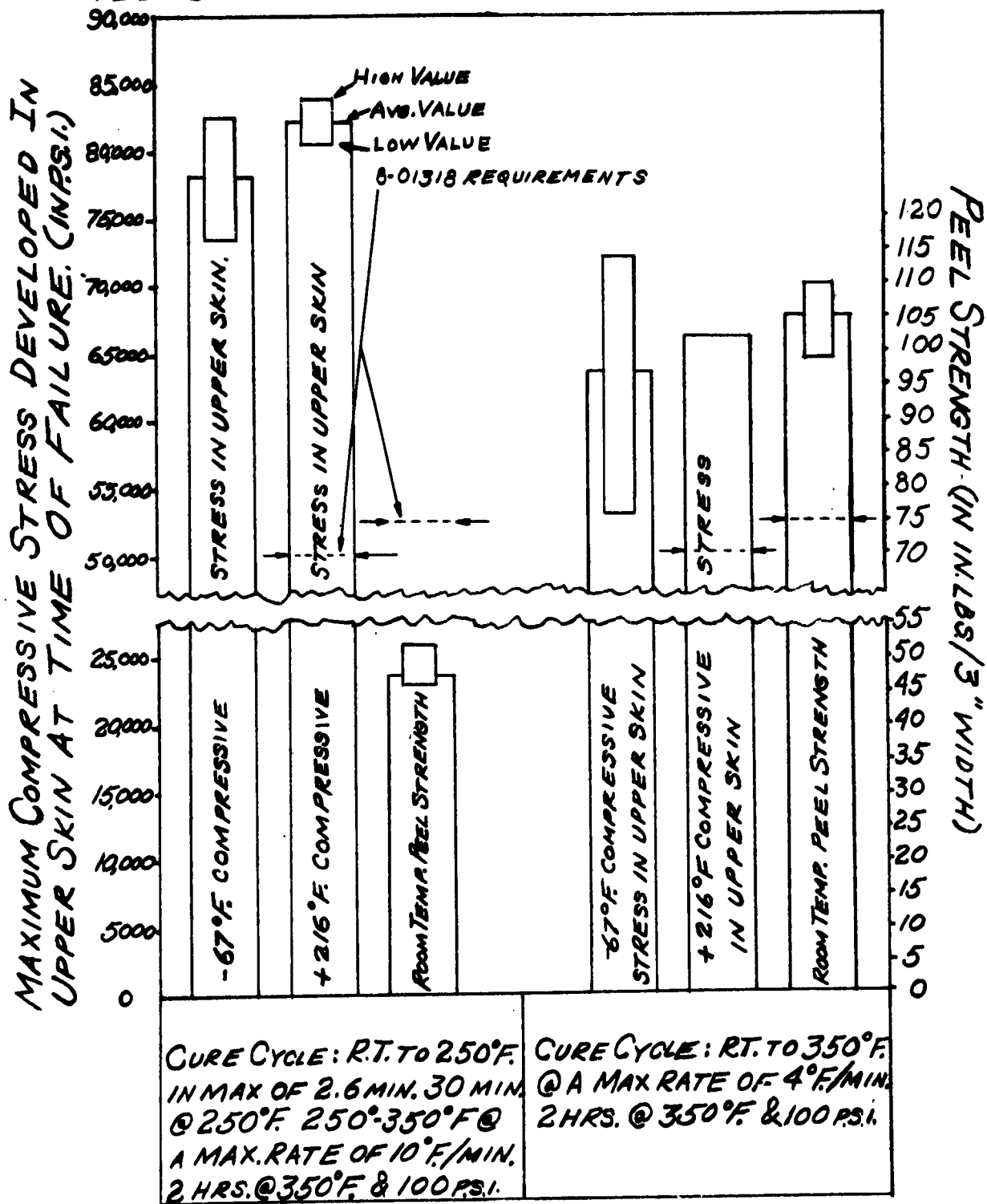
A bar chart titled 'SPECIFICATION REQUIREMENT * (0-03007)'. The vertical axis (y-axis) is labeled with values from 3200 to 4000 in increments of 200. The chart displays a single bar with a height of approximately 3450. Three arrows point to the bar: 'HIGH VALUE' points to the top of the bar, 'AVERAGE VALUE' points to the middle of the bar, and 'LOW VALUE' points to the bottom of the bar.



* 216°F REQUIREMENT
ESTIMATED BY INTERPOLATING
R.T. & +300°F SPEC. REQUIREMENTS

FIG II

EFFECTS OF TWO CURE CYCLES ON THE -67° & $+216^{\circ}$ F. FLEXURAL STRENGTHS, AND THE ROOM TEMPERATURE PEEL STRENGTHS OF SANDWICH HONEYCOMB PANELS.



CURECYCLES INVESTIGATED.

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TABLE I

METAL TO METAL BONDS WITH AF-31 ADHESIVE

TENSILE SHEAR RESULTS

TEST TEMP °F.	CURE CYCLE INVESTIGATED	TENSILE SHEAR PSI.	AVG. T.S. PSI.	TYPE FAILURE
-67°	HEAT FROM ROOM TEMP. TO 350°F. IN 25 MINUTES. CURE FOR 2 HOURS AT 350°F WITH 150 PSI PRESSURE. (TEMP. RISE 11°F/MIN.)	3090	3303	BETWEEN
-67°		3482		PRIMER
-67°		3340		& ADHESIVE
+216°		2720	2793	COHESIVE
+216°		2880		"
+216°		2780		"

PEEL RESULTS

TEST TEMP °F.	CURE CYCLE INVESTIGATED	PEEL STRENGTH LB./IN. WIDTH	AVG. PEEL STRENGTH	TYPE FAILURE
+75°	SAME AS ABOVE	11.6	10.2	COHESIVE
+75°		9.7		"
+75°		9.7		"
+75°		9.7		"

MATERIALS:

1. ADHESIVES: E.C. 1459 PRIMER & AF-31 TAPE
2. ADHERENDS:
 - A. TENSILE SHEAR SPECIMENS: .063" x 4" x 7" 2024 T3 CLAD AL. ALLOY; MILLED EDGE PANELS CUT INTO 1" x 7 1/2" SPECIMENS WITH 1/8" OVERLAP.
 - B. PEEL SPECIMENS: .020" x 1" x 9" 2024 T86 CLAD AL. ALLOY STRIPS.

ANALYSIS
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TABLE II
 METAL TO METAL BONDS WITH AF-31 ADHESIVE
 TENSILE SHEAR RESULTS

TEST TEMP °F.	CURE CYCLE INVESTIGATED	TENSILE SHEAR PSI.	AVG. T.S. PSI.	TYPE FAILURE
-67°	ROOM TEMP. TO 250°F. IN A MAX. OF 2.6 MIN. 30 MIN. @ 250°F.	2610	2367	PRIME.
-67°		2200		PRIME & AD.
-67°		2290		PRIME
+216°	250°-350°F. @ A MAX. RATE OF 10°F/MIN. CURE FOR 2 HRS. @ 350° F. AND 100 PSI	2370	2330	COHESIVE
+216°		2400		"
+216°		2220		"

PEEL RESULTS

TEST TEMP °F.	CURE CYCLE INVESTIGATED	PEEL STRENGTH LBS./IN. WIDTH	AVG. PEEL STRENGTH	TYPE FAILURE
+75°	SAME AS ABOVE	9.6	11.1	COHESIVE
+75°		11.6		"
+75°		11.6		"
+75°		11.6		"

MATERIALS:

1. ADHESIVES: E.C. 1459 PRIMER & AF-31 TAPE
2. ADHERENDS:

A. TENSILE SHEAR SPECIMENS: .063" X 4" X 7" 2024 T3
 CLAD AL. ALLOY; MILLED EDGE PANELS CUT INTO
 1" X 7½" SPECIMENS WITH ½" OVERLAP.

B. PEEL SPECIMENS: .020" X 1" X 9" 2024 T86 CLAD
 AL. ALLOY STRIPS

ANALYSIS

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TABLE III

METAL TO METAL BONDS WITH AF-31 ADHESIVE
TENSILE SHEAR RESULTS

TEST TEMP °F.	CURE CYCLE INVESTIGATED	TENSILE SHEAR PSI.	AVG T.S. PSI.	TYPE FAILURE
-67°	HEAT FROM ROOM TEMPERATURE TO 350° F. AT A MAX. RATE OF 4° F/MIN. CURE FOR 2 HRS. AT TEMP. WITH 100 PSI. PRESS- URE.	1850	1903	BETWEEN PRIMER & ADHESIVE
-67°		1980		
-67°		1880		
+216°		2240	2287	COHESIVE
+216°		2200		"
+216°		2420		"

PEEL RESULTS

TEST TEMP °F.	CURE CYCLE INVESTIGATED	PEEL STRENGTH LBS/IN. WIDTH	AVG. PEEL STRENGTH	TYPE FAILURE
+75°	SAME AS ABOVE	17.4	16.4	COHESIVE
+75°		17.4		"
+75°		15.4		"
+75°		15.4		"

MATERIALS:

1. ADHESIVES: E. C. 1459 PRIMER & AF-31 TAPE

2. ADHERENDS:

A. TENSILE SHEAR SPECIMENS: .063" X 4" X 7" 2024 T3
CLAD AL. ALLOY, MILLED EDGE PANELS CUT INTO
1" X 7 1/2" SPECIMENS WITH 1/2" OVERLAP.B. PEEL SPECIMENS: .020" X 1" X 9" 2024 T86 CLAD
AL. ALLOY STRIPS.

TABLE IV
 METAL TO METAL BONDS WITH AF-31 ADHESIVE
 TENSILE SHEAR RESULTS

TEST TEMP °F	CURE CYCLE INVESTIGATED	TENSILE SHEAR, PSI	AVG T.S. PSI	TYPE FAILURE
-67°	ROOM TEMP TO 350°F AT A MAX RATE OF 4°F./MIN. CURE FOR 2 HRS. AT TEMP WITH 85 PSI. PRESSURE.	1760	1807	PRIMER & ADHESIVE.
-67°		1950		
-67°		1710		
+216°		2480	2373	COHESIVE
+216°		2320		"
+216°		2320		"

PEEL RESULTS

TEST TEMP °F	CURE CYCLE INVESTIGATED	PEEL STRENGTH LBS./IN WIDTH	AVG PEEL STRENGTH	TYPE FAILURE
+75°	SAME AS ABOVE	17.4	18.0	COHESIVE
+75°		17.4		"
+75°		19.3		"
+75°		17.4		"

MATERIALS:

1. ADHESIVES: E.C. 1459 PRIMER & AF-31 TAPE.

2. ADHERENDS:

A. TENSILE SHEAR SPECIMENS: .063" X 4" X 7" 2024 T3 CLAD AL. ALLOY, MILLED EDGE PANELS CUT INTO 1" X 7½" SPECIMENS WITH ½" OVERLAP.

B. PEEL SPECIMENS: .020" X 1" X 9" 2024 T86 CLAD AL. ALLOY STRIPS.

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TABLE V

SANDWICH HONEYCOMB FLEXURAL & PEEL TEST RESULTS

FLEXURAL RESULTS

TEST TEMP. °F	CURE CYCLE INVESTIGATED	"A" DISTANCE	CORE SHEAR PSI.	COMPRESSIVE STRESS PSI.	TYPE FAILURE
-67°	ROOM TEMP. TO 250°F. IN A MAX. OF 2.6 MIN. 30 MIN. @ 250°F. 250°-350°F. @ A MAX. RATE OF 10°F/MIN. CURE FOR 2 HRS. @ 350°F. AND 100 PSI. PRESSURE.	2.14"	657	72,900	CORE SHEAR & ADHESIVE.
-67°		"	774	82,500	
-67°		"	726	78,000	
+216°		2.52"	636	81,000	CORE SHEAR
+216°		"	666	83,800	

PEEL RESULTS

TEST TEMP. °F.	CURE CYCLE INVESTIGATED	PEEL STRENGTH IN LBS/3" WIDTH	AVG. PEEL STRENGTH	TYPE FAILURE
+75°	SAME AS ABOVE	52.1	47.5	BETWEEN FIBERGLAS REINFORCEMENT & ADHESIVE
+75°		46.3		
+75°		46.3		
+75°		46.3		
+75°		46.3		

MATERIALS:

1. ADHESIVES:

- a. PRIMER: E.C. 1459
- b. LIQUID ADHESIVE: E.C. 1469
- c. FILM ADHESIVE: A.F. 102

2. ADHERENDS:

- a. SKINS: .020" 2024 T86 CLAD AL. ALLOY
- b. CORE: .375" 3003 H19 AL. ALLOY .002" FOIL, 1/8" HEX CELL.

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TABLE VI

SANDWICH HONEYCOMB FLEXURAL & PEEL TEST RESULTS

FLEXURAL RESULTS

TEST TEMP. °F.	CURE CYCLE INVESTIGATED	"A" DISTANCE	CORE SHEAR P.S.I.	COMPRESSIVE STRESS P.S.I.	TYPE FAILURE
-67°	HEAT FROM ROOM	2.14"	680	72,200	PERMANENT
-67°	TEMP. TO 350°F. AT A	"	615	65,300	SET IN
-67°	MAX. RATE OF 4°F./	"	494	52,800	SANDWICH
+216°	MIN. CURE FOR 2 HRS.	2.52"	528	66,500	CORE SHEAR
+216°	AT TEMP. WITH 100	"	528	66,500	& UPPER SKIN BUCKLING.
+216°	P.S.I. PRESSURE.	"			

PEEL RESULTS

TEST TEMP. °F.	CURE CYCLE INVESTIGATED	PEEL STRENGTH IN LBS./3" WIDTH	AVG. PEEL STRENGTH	TYPE FAILURE
+75°	SAME AS ABOVE	98.4	105	BETWEEN FIBERGLAS REINFORCEMENT & ADHESIVE.
+75°		104		
+75°		104		
+75°		110		
+75°		110		

MATERIALS:

1. ADHESIVES:

a. PRIMER: EC 1459

b. LIQUID ADHESIVE: E.C. 1469

c. FILM ADHESIVE: AF 102

2. ADHERENDS:

a. SKINS: .020" 2024 T86 CLAD AL. ALLOY

b. CORE: .375" 3003 H19 AL ALLOY .002" FOIL, 1/8" HEX CELL.